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New effect pigments based on SiO₂ and Al₂O₃ flakes

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Stephen Sharrock joined Merck in 1973 after graduating from Loughborough University, England. He moved from the Laboratory Reagents Division of Merck to the Pigments Division in 1982 and has been working in Marketing & Sales for Automotive Pigments since 1988, where today he is Group Leader. He is furthermore responsible for the marketing side of the SiO₂ and Al₂O₃ flake projects.

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Norbert Schuel joined Merck in 1983 after graduating in Mechanical Engineering at Darmstadt. Initially, he worked in the Central Process Development Department where he was involved in several technical projects, both in the chemical and pharmaceutical areas. In 1991 he was assigned to R&D projects from the Pigments Division, including the development of SiO₂ flakes. He later moved to the Pigment Division and became Project Leader for the SiO₂ flakes. Today he is also Production Plant Manager for the SiO₂ flakes.

Pigments based on thin mica platelets coated with metal oxides are important types of pearlescent pigments today. They show many advantages, e.g. excellent interference colours and good application behaviour. Two new synthetic materials can now be used, besides mica, as substrates for effect pigments. New colouristic effects can be realized by coating the silica or alumina flakes with highly refractive metal oxides such as titanium dioxide or iron oxide. The theory, production and properties of the new effect pigments were already discussed. In this new paper the results of technical work on the incorporation, application and colour formulation of the new effect pigments are presented.

Pearlescent Pigments are used to achieve vivid interactions of colour and luster with the added dimension of variation depending on the angle of illumination and observation. These pigments play an important role in the creation of many new and unique effects when used in plastics, printing inks, cosmetic formulations and paints for automotive and industrial application. The unique pearlescent effect is produced by light interference rather than absorption. However, they compliment and enhance the appearance created by conventional absorption and/or metal effect pigments [1-3]. Special colour effects, chemical stability, and ease of use are their main advantages. Furthermore, they are not toxic. Pigments based on the layer-substrate interference principle are the most important types of pearlescent pigments today. Most of them consist of metal oxide layers coated on mica, e.g. "Iridin" pigments [1-5]

**New synthetic
substrates yield
new effect
pigments**

Several years ago, Merck began investigating new synthetic materials that could be used in place of mica as substrates for effect pigments. The objective was to develop additional varieties that would exhibit the well-known advantages of mica pigments (eg. good application properties in a variety of binder systems, environmental compatibility and simple handling) with the possibility of realizing unique optical effects. Two of these new substrates are silica flakes [6] and

alumina flakes [7]. As synthetic materials, they permit degrees of design freedom that were previously unavailable, and when coated with metal oxides they can be used for generating new effects that extend the product variety and performance range of pearlescent pigments [8]. The silica flakes are absolutely flat, and in combination with selection of thickness to optimize design, more intense interference colours with a variety of gonio-colourimetric properties are feasible. Natural substrates have a much wider distribution of thickness.

The SEM micrograph in [Figure 1](#) shows a cross section through a silica flake coated with $\alpha\text{-Fe}_2\text{O}_3$ (Hematite). It shows clearly that both the SiO_2 flake and the Fe_2O_3 layer thickness are precisely controlled. The new effect pigments based on SiO_2 are available under the tradename "Colorstream".

The corundum modification of Al_2O_3 crystallises as hexagonal closest packing. When the crystal grows perfectly along the c-axis, the atomic structure will layer uniformly, thereby generating a very flat surface to the flakes.

The introduction of contaminants can be eliminated or controlled by the crystal growth process, furthermore the production process provides a narrow particle size and thickness distribution. The SEM micrograph in [Figure 2](#) shows

Al_2O_3 -flakes with such properties which are the basis for strongly reflecting pigments when coated with high refractive index metal oxides. The new effect pigments based on Al_2O_3 flakes are available under the tradename "Xirallic".

Incorporation, application and colour

formulation with the new pigments

Iron oxide coated SiO_2 Flakes

The new effect pigments disperse well into both solvent and waterborne formulations and spray out the same as current pearlescents using identical application parameters.

The colour formulation of 2-coat and 3-coat automotive colours is also very similar to mica pigments, however, the following points should be observed:

- Good results are achieved if - in comparison to Fe_2O_3 / mica pigments - lower pigmentations are chosen. A pigmentation of 0.7 to 1.5 % (based on the total paint at spray viscosity) is recommendable, but higher pigmentations are of course possible.
- Caution is advisable when using this type of pigment with metallic pigments. Too much metallic pigment can lead to a loss of colour travel.
- Just like for mica pigments, caution is also advisable when using this type of pigment in combination with opaque or semi-opaque conventional pigments. If the pigmentation level of these pigments is too high a loss of colour travel is again possible.

Table 1 shows a typical formulation for a 2-coat colour using a Fe_2O_3 / SiO_2 pigment.

Table 1: Formulation for a 2-coat colour with a colour travel from green to blue to lilac

2.00 %	Fe ₂ O ₃ / SiO ₂ pigment (EM 142537)
1.50 %	Phthalocyanine Blue P.B. 15:1 / 74160
0.20 %	Phthalocyanine Green P.G.36 / 74265
0.05 %	Carbon Black P.Bk. 7 / 77266

Solid content (formulation)	16.41 %
P/B	30/100
Styling No.	EN 67/98-9

TiO₂ coated SiO₂ Flakes

- This type of pigment is very transparent. Compared to TiO₂ / mica types slightly higher pigmentation levels of 2-3% are advisable.
- These pigments are also very sensitive towards combinations with metallic pigments and opaque or semi-opaque conventional pigments. Again caution is advisable with regard to the pigmentation levels.
- These pigments are very suitable for styling attractive 3-coat colours in the pastel area. Very interesting colour travel effects combined with a silvery hue are possible.

Table 2 shows a typical formulation for a 2-coat colour using a TiO₂ / SiO₂ pigment.

Table 2: Formulation for a 2-coat colour with a colour travel from blue-violet to green

2.00 %	TiO ₂ / SiO ₂ pigment (EM 142529)
1.00 %	Phthalocyanine Green P.G. 36 / 74265
1.00 %	Indanthrone Blue P.B. 60 / 69800
0.30 %	Transparent iron oxide yellow P.Y.42 / 77492
0.05 %	Carbon Black P.Bk. 7 / 77266

Solid content (formulation)	16.50 %
P/B	30/100
Styling No.	MIP 178/98

Al₂O₃ Flake pigments

Colour formulation is basically identical to comparable mica pigments. A pigmentation level of 1-2.5 % is ideal. The degree of sparkle which the styling shows can also be controlled by the pigmentation level. With regard to combinations with metallic and conventional colour pigments Al₂O₃ flake pigments behave in the same way as comparable mica pigments.

Combination is feasible

It is of course possible to combine both SiO_2 flake and Al_2O_3 flake pigments when styling colours. Likewise combinations with mica are also possible. The virtually unlimited styling possibilities are a rewarding challenge to the colour designer.

Technical performance of the new pigments in automotive paints

Performance evaluations in OEM systems (with a P/B as high as 0.35) indicate excellent adhesion properties and retention and recovery of both DOI and gloss in humidity tests.

Table 3 shows results for various pigments in a condensed water test in an OEM waterborne paint system.

Table 3: Condensed water test results according to DIN 50017 (40 °C, 240h) in an OEM waterborne paint system

Pigment	Before test	Evaluation 1h after test completion				
		Blistering	Swelling	Rem. Dol in %	Rem. gloss in %	Cross-hatch
$\text{TiO}_2/\text{SiO}_2 + \text{SW}$	OK	m0g0	Q 0	100	100	Gt 0-1
$\text{Fe}_2\text{O}_3/\text{SiO}_2 + \text{SW}$	OK	m0g0	Q 2	85	100	Gt 1
$\text{TiO}_2/\text{Al}_2\text{O}_3$ Silver SW	OK	m0g0	Q 0-1	93	100	Gt 0-1
$\text{Fe}_2\text{O}_3/\text{Al}_2\text{O}_3$ Red SW	OK	m0g0	Q 0	93	100	Gt 1

Table 4 shows results for the same pigments in Xenotest 1200 accelerated weathering in a conventional (solvent-borne) 2-coat OEM paint system.

Table 4: Xenotest 1200 results in a solvent-borne 2-coat OEM paint system

Pigment	1000 h		2000 h	
	Grey scale	Remaining gloss in %	Grey scale	Remaining gloss in %
$\text{TiO}_2/\text{SiO}_2 + \text{SW}$	4-5	91	4-5	73
$\text{Fe}_2\text{O}_3/\text{SiO}_2 + \text{SW}$	4	95	4-3	78
$\text{TiO}_2/\text{Al}_2\text{O}_3$ Silver SW	5-4	100	4	88
$\text{Fe}_2\text{O}_3/\text{Al}_2\text{O}_3$ Red SW	4-5	100	4	100

Thickness of the flake and chemical purity expand the range of possibilities

The new generations of effect pigments, based on synthetic silica and alumina flakes coated with highly refractive metal oxides, expand the range of possibilities for stylists and designers in different application fields. Silica and alumina flake pigments with their precisely defined and controlled properties, especially thickness of the flake and chemical purity, open the door to a new era of engineered pigment colouristics. We look forward to exploiting these capabilities to provide a new interactive resource for innovation and imagination in areas such as automotive coatings, coloured plastics, printing inks, ceramic products and cosmetic formulations.

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"Iridin", "Colorstream" and "Xirallic" are registered trade names of Merck KGaA, Darmstadt, Germany

Tables

- Table 1: Formulation for a 2-coat colour with a colour travel from green to blue to lilac
- Table 2: Formulation for a 2-coat colour with a colour travel from blue-violet to green
- Table 3: Condensed water test results according to DIN 50017 (40 °C, 240h) in an OEM waterborne paint system
- Table 4: Xenotest 1200 results in a solvent-borne 2-coat OEM paint system

Figures

- Figure 1 Cross-section through a silica flake coated with $\alpha\text{-Fe}_2\text{O}_3$ (Hematite)
- Figure 2 Electron micrograph of Al_2O_3 flakes